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EVALUATION OF TWO NICKEL-BASE ALLOYS, ALLOY 713C AND NASA TAZ-8A, PRODUCED BY EXTRUSION OF PREALLOYED POWDERS

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ABSTRACT

Prealloyed powders of Alloy 713C and TAZ-8A, were made by inert gas atomization and extruded. The powder products were evaluated by tensile and stress rupture tests in the as-extruded and heat-treated conditions. Significant improvements in room temperature and 1200° F (649° C) strength were obtained over the as-cast condition but lower strengths and superplastic behavior were observed for the extruded powder alloys in the 1800° to 2000° F (982° to 1094° C) temperature range. It was possible to hot form the TAZ-8A powder product at low strain rates and low applied loads. Heat treatments eliminated superplasticity.

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SUMMARY

Prealloyed powders of two nickel-base alloys, Alloy 713C and NASA TAZ-8A, were made by inert gas atomization and extruded into bar stock. The powder products were evaluated in the as-extruded condition and after various heat treatments by tensile and stress rupture tests.

Tensile strength of the as-extruded powder products was substantially greater than that of conventionally cast or wrought nickel-base alloys up to 1400° F (760° C). Room temperature ultimate tensile strengths for Alloy 713C and TAZ-8A as-extruded powder products were 221 700 pounds per square inch (1530 MN/m^2) and 228 000 psi (1570 MN/m^2), respectively, as compared to 123 000 psi (846 MN/m^2) and 128 000 psi (882 MN/m^2) in the as-cast condition. In the 1800° to 2000° F (982° to 1094° C) range the as-extruded powder product of both alloys had lower tensile and rupture strengths than cast alloys and exhibited very high neck-free elongations; (e.g., over 230 percent after testing at 2000° F (1094° C) and 2000 psi (13.8 MN/m^2) for Alloy 713C, and over 600 percent after testing at 1900° F (1038° C) and 1000 psi (6.89 MN/m^2) for TAZ-8A). These large amounts of strain suggest superplastic behavior. It was possible to take advantage of the superplastic behavior of TAZ-8A by hot pressing at low loads and low strain rates.

The extruded powder products of both alloys had substantially greater rupture lives at 1200° F (649° C) and 105 000 psi (724 MN/m^2) than a strong conventional wrought alloy. However, in the 1800° to 2000° F (982° to 1094° C) temperature range, rupture lives of both alloys were considerably lower than in the as-cast condition.

The simultaneous application of pressure and temperature made it possible to heat treat the TAZ-8A powder product above the incipient melting point without void formation. This suggests a possible means of achieving the coarseness of microstructure needed to improve high temperature strength.

INTRODUCTION

Conventionally cast and wrought nickel-base alloys continue to be the workhorse materials for the hot components of gas-turbine engines. The cast alloys are generally used for turbine buckets and stator vanes whereas wrought alloys are used for turbine disks, and, in more advanced engines, for compressor disks and blades in the latter compressor stages. To meet the demand for increased performance, designers of advanced engines must raise the operating cycle temperature. It therefore remains an important objective to provide nickel-base alloys that can be used at higher temperatures throughout the engine.

Unfortunately, most high-strength, nickel-base alloys are highly alloyed and metallurgically very complex. As a consequence, severe macro- and micro-segregation can occur in castings such as turbine buckets and stator vanes so that the full-strength potential of the alloy is not realized. In ingots, the usual starting stock for breakdown operations, segregation increases the difficulty of forming the alloys. Indeed many highly alloyed compositions with high as-cast properties cannot be worked by normal procedures.

The use of prealloyed powders affords a means of overcoming the problems inherent in conventional casting and hot-working operations of superalloys. By atomizing the molten alloy with an inert gas jet, each metal droplet is subjected to rapid solidification rates. This results in homogeneous powder particles and a homogeneous structure upon subsequent compaction of the powder. Use of inert gas in the atomization process minimizes the oxygen content of the powders. This, in turn, militates against the formation of large oxide particle inclusions and facilitates powder compaction by preventing the formation of tightly adherent oxide films on the powder particles.

Other investigators (refs. 1 and 2) have employed the general concept of prealloyed powders to produce wrought nickel-base superalloy products with promising results. Limited data from reference 1 show about a twofold improvement in room temperature and 2000° F (1094° C) tensile strength for Alloy 713C extruded powder product over the cast alloy. The reference also shows that the 100-hour, 1800° F (982° C) stress-rupture strength is about twice as high as that of the cast alloy. The data of reference 2 show that extruded Udimet 700-powder product had greater room-temperature tensile strength, approximately equal 1400° F (760° C) strength, and lower 1800° F (982° C) strength than conventionally cast or cast-and-wrought Udimet 700.

The purpose of the present investigation was to apply the prealloyed powder concept to two cast nickel-base alloys in order to obtain improved strength and ductility over a wide range of temperatures. A commercial cast alloy, Alloy 713C, and a promising experimental cast alloy, TAZ-8A (ref. 3), were chosen. Bars extruded from high-purity powders made by inert-gas atomization were purchased from a commercial alloy manufacturer. Tensile and stress rupture tests were run with as-extruded material. The effect of various heat treatments on the properties of the extruded powder product was also evaluated by tensile and stress rupture tests. In addition, metallographic analyses were made. Data comparisons are presented with conventionally produced material for both alloys.

MATERIALS AND PROCEDURE

Materials

Bars of two nickel-base superalloys, Alloy 713C and TAZ-8A, extruded from prealloyed powders were obtained from a commercial alloy manufacturer. The average Rockwell C hardness of the bars was 43.5 for Alloy 713C and 51.5 for TAZ-8A. The alloys were melted under argon in a 75-pound (34-kg) capacity induction furnace and atomized under argon to spheroidal powders. The powders were screened with Tyler screens to -60 mesh and only the -60 mesh fraction was used for the extrusions. The sieve analyses for the -60 mesh fraction for each alloy are given in table I. The photo micrographs in figure 1 show the spheroidal shape of the powder particles from representative samples of both of the alloys. Some porosity is evident in the powder particles, and the powder particles of both alloys exhibited a dendritic structure. The massive segregation encountered in the as-cast alloys has been essentially eliminated in the powder particles.

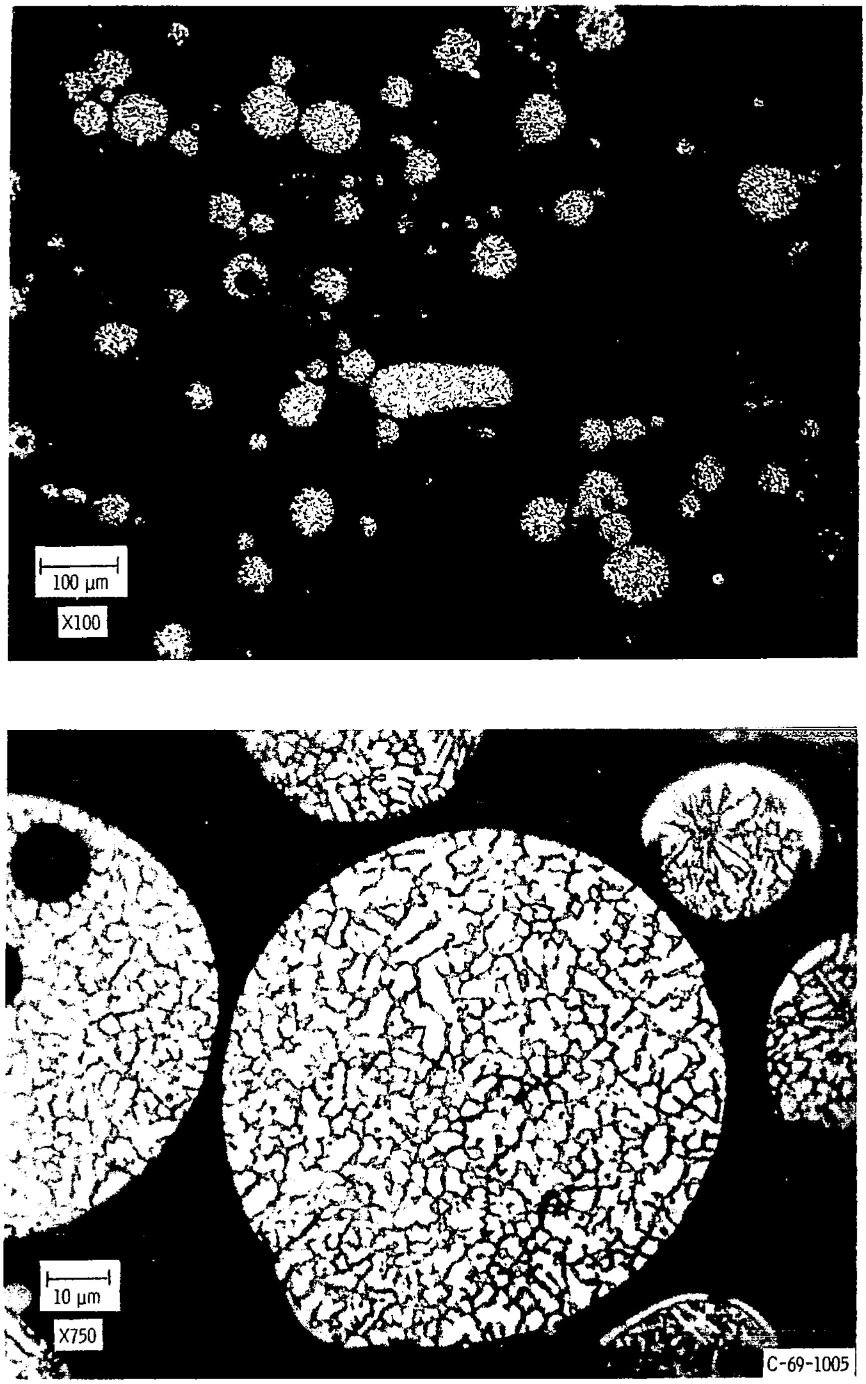
The powders were sealed in evacuated mild steel cans and extruded directly into bars approximately 9/16 inch (1.43 cm) in diameter. The cans had a $2\frac{3}{8}$-inch (6-cm) outside diameter and a 2-inch (5-cm) inside diameter. Extrusions were made through a 7/8-inch (2.2-cm) diameter die having a 90° included angle. The $2\frac{1}{2}$-inch (6.3-cm) inside diam-

TABLE I. - PARTICLE SIZE DISTRIBUTION

OF ATOMIZED POWDERS[a]

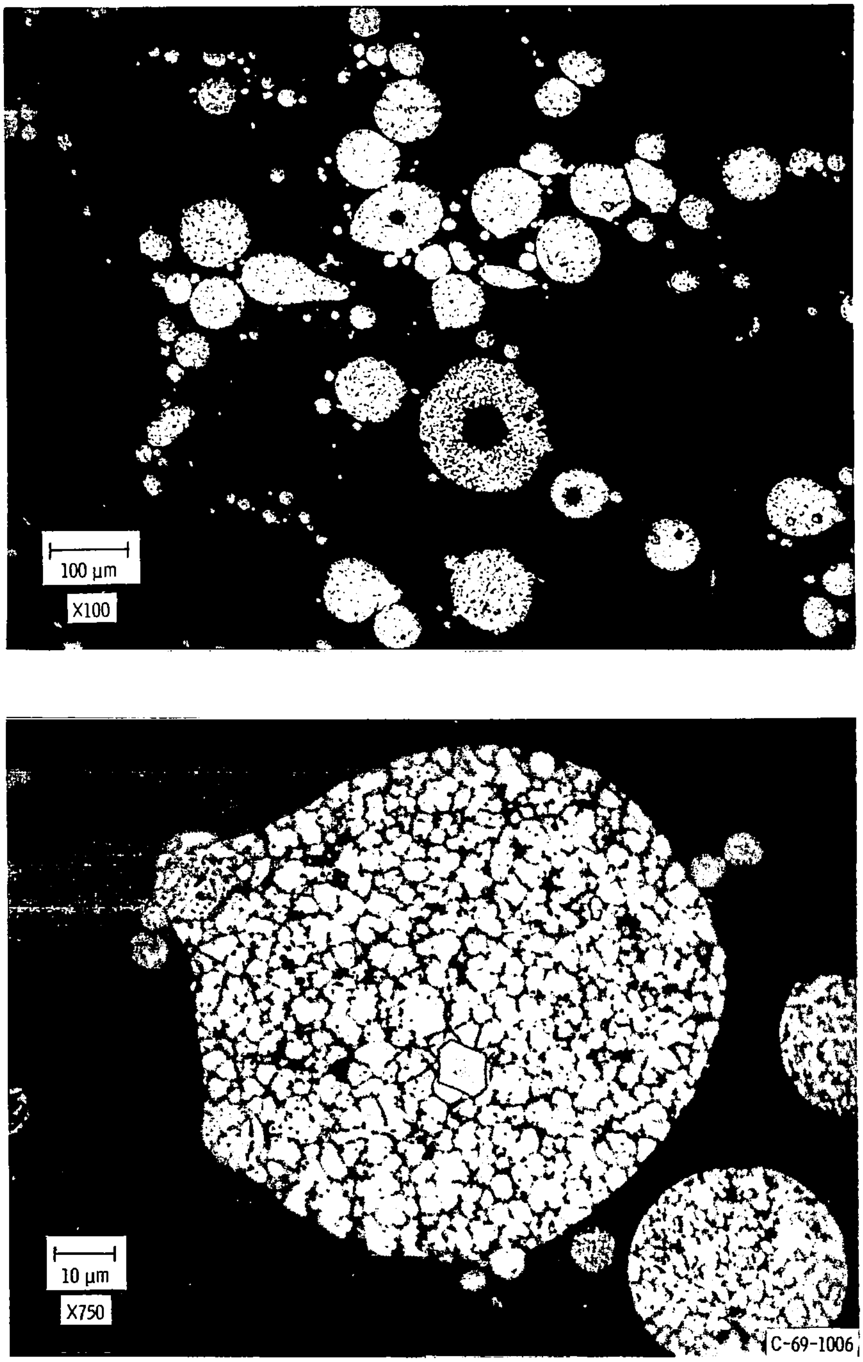
Tyler screen size	Particle size distribution, percent	
	Alloy 713C	TAZ-8A
60/100	7.5	5.0
100/500	14.5	13.5
150/270	30.0	30.0
270/325	5.5	7.0
325/400	8.5	9.0
<400	33.5	35.5

[a]Vendor's analysis.



(a) Alloy 713 C.

Figure 1. - As-received powders of Alloy 713 C and TAZ-8A.



(b) TAZ-8A.

Figure 1. - Concluded.

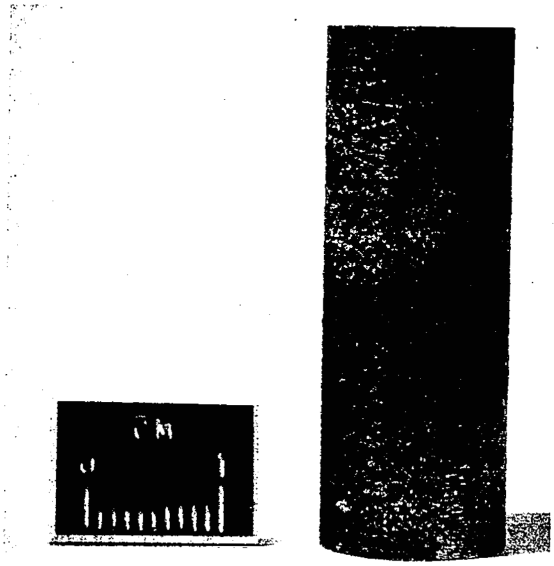


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Figure 2. - Surface of typical Alloy 713 C extrusion after removal of can.

eter of the chamber provided an extrusion ratio of approximately 8.2 to 1. The canned powders were heated to 2200° F (1204° C) in a furnace prior to extrusion. About 10 seconds were required to transfer the sample from the furnace to the extrusion press.

Figure 2 shows the surface of a typical Alloy 713C extrusion after the cans were chemically removed. Since the surface of the bars was rough, they were ground to permit fluorescent dye penetrant inspection. No cracks were observed. The bars were also inspected radiographically with a minimum of 2 percent sensitivity. The Alloy 713C was radiographically sound, but approximately 60 percent of the length of the extruded TAZ-8A bars was rejected for indications of flaws similar in appearance to centerline shrinkage in a cast bar. This may be an indication of center burst. Extrusion at a higher temperature might have resulted in a better extruded product.

The interiors of the bars were dense, except for occasional tearing in TAZ-8A. However, near the surface of the bars of both alloys complete bonding of the powders was not achieved. In polished sections the outlines of individual powder particles could still be seen. Random density measurements of the extruded Alloy 713C and TAZ-8A powder products gave values of 7.96 and 8.55 grams per cubic centimeter. These values compared to 7.91 grams per cubic centimeter reported for cast Alloy 713C and 8.65 grams per cubic centimeter for cast TAZ-8A. The differences in the measured densities between the cast and extruded powder products may be due to slight differences in alloy chemistries or microporosity in the samples measured.

The vendor's chemical analyses of both alloys made from samples of the powder and from extruded bars are listed in table II. The table also includes gas analyses of ex-

TABLE II. - CHEMICAL ANALYSES OF ALLOYS USED

Element	Alloy 713C			TAZ-8A		
	Specified composition	Powder	Extrusion	Specified composition	Powder	Extrusion
	Element, wt. %					
Tantalum	------	------	-------	8	8.17	7.72
Tungsten	------	------	-------	4	4.00	3.93
Molybdenum	4.5	4.49	4.57	4	3.99	4.02
Columbium	------	------	-------	2.5	2.67	2.49
Columbium and tantalum	2	2.42	2.30	-----	-----	-------
Chromium	14	13.55	13.33	6	6.10	6.17
Aluminum	6	5.78	5.26	6	6.08	5.68
Titanium	1	.66	.64	-----	-----	-------
Zirconium	.10	.08	.14	.75	.62	.77
Boron	.01	.012	.015	-----	.006	.005
Carbon	[a].2	.14	.14	.125	.15	.17
Iron	[a]3.0	.80	.70	-----	.13	-------
Cobalt	[a]1.0	.42	.67	-----	.36	-------
Manganese	[a]1.0	.05	.05	-----	.03	-------
Phosphorous	------	.002	.002	-----	.002	.002
Sulfur	[a].015	.003	.004	-----	.004	.006
Silicon	[a]1.0	.15	.18	-----	.04	.10
Nitrogen	------	.008	[b].0079	-----	.018	[b].0087
Oxygen	------	.016	[b].0170	-----	.013	[b].013
	------	------	[b].0113	-----	-----	[b].0150
Nickel	Balance	Balance	Balance	Balance	Balance	Balance

[a]Maximum.
[b]By independent laboratory.

truded bar samples made by an independent laboratory. For both alloys, the powder came very close to the specified compositions. Except for aluminum content, which was 5.26 percent rather than 6 percent for the extruded Alloy 713C, the extruded powder products also came close to the specified compositions. Also, the oxygen content of the extruded bars was less than 150 ppm, while the total oxygen plus nitrogen content did not exceed 240 ppm. The aim composition used for Alloy 713C is from an aircraft engine manufacturer's specification. The aim composition for TAZ-8A is from reference 3.

Heat Treating

Heat treatments to effect solutioning and aging were performed in vacuum or under

TABLE III. - HEAT TREATMENTS APPLIED TO EXTRUDED ALLOY 713C

AND TAZ-8A POWDER PRODUCTS

Heat treatment[a]				Designation
Step 1	Step 2	Step 3	Step 4	
Alloy 713C				
4 hr at 2250° F (1232° C); furnace cooled[b]	4 hr at 2000° F (1094° C); air cooled	24 hr at 1500° F (816° C); air cooled	16 hr at 1400° F (760° C); air cooled	A
4 hr at 2250° F (1232° C); air cooled	4 hr at 2000° F (1094° C); air cooled	24 hr at 1500° F (816° C); air cooled	16 hr at 1400° F (760° C); air cooled	B
4 hr at 2250° F (1232° C); furnace cooled[b]	--------------	--------------	--------------	C
TAZ-8A				
4 hr at 2350° F (1288° C); air cooled	4 hr at 2000° F (1094° C); air cooled	24 hr at 1500° F (816° C); air cooled	16 hr at 1400° F (760° C); air cooled	X
1/2 hr at 2350° F (1288° C); air cooled	--------------	--------------	--------------	Y
1/2 hr at 2400° F (1316° C); air cooled	--------------	--------------	--------------	Z
Autoclave: 2 hr at 2400° F (1316° C) and 10 000 psia (68.9 MN/m^2); furnace cooled[c]	--------------	--------------	--------------	Q

[a]In argon except where noted.
[b]In vacuum.
[c]In helium.

argon on unmachined extruded bars of both alloys. The heat treatments used for each alloy are listed in table III. Each has been given a letter designation to simplify subsequent referencing. Because it was impossible to cause appreciable grain coarsening or solutioning of TAZ-8A without incipient melting, a heat treatment was carried out in an autoclave under a helium pressure of 10 000 psi (68.9 MN/m^2). Furnace temperature variations of ±10° F (±6° C) were observed in the atmosphere heat treating furnaces. Variations of ±30° F (±17° C) were observed in the autoclave.

Mechanical Testing

Tensile and stress rupture tests were made in air at temperatures up to 2000° F (1094° C) with as-extruded and with heat-treated material. The test conditions and data

TABLE IV. - TENSILE DATA

Condition	Test temperature		Ultimate tensile strength		Elongation, percent
	°F	°C	psi	MN/m^2	
Alloy 713C					
As-extruded	Room	Room	[a]221 700	1530	22
	1200	649	194 900	1340	5.5
	1400	760	153 800	1060	6
	1800	982	[a]43 200	298	8
	2000	1094	13 800	95	45
Heat treatment B	Room	Room	169 300	1170	13
	1200	649	162 000	1120	16
	1400	760	141 000	973	13
	1800	982	57 800	398	7
	2000	1094	23 200	160	9
TAZ-8A					
As-extruded	Room	Room	[a]228 000	1570	5
	1200	649	218 200	1500	3.5
	1400	760	164 400	1130	3.3
	1800	982	[a]7 100	49	>450
Heat treatment X	Room	Room	142 200	980	2
	1200	649	166 500	1150	3
	1400	760	154 000	1060	3
	1800	982	48 600	335	3
Heat treatment Y	1800	982	51 100	352	--------

[a]Test conducted by independent laboratory.

TABLE V. - STRESS RUPTURE DATA

Condition	Test temperature		Stress		Life, hr	Elongation, percent
	°F	°C	psi	MN/m^2		
Alloy 713C						
As-extruded	1200	649	105 000	724	538.1	3
	1900	1038	[a]5 000	34.5	6.3	162
	1900	1038	[a]10 000	68.9	1.2	60
	2000	1094	[a]2 000	13.8	7.6	230
	2000	1094	[a]10 000	68.9	.2	42
Heat treatment C	1900	1038	10 000	68.9	98.1	4
Heat treatment A	1900	1038	10 000	68.9	1.5	39.5
Heat treatment B	1200	649	105 000	724	761.2	3
	1200	649	105 000	724	462.7	<1
	1800	982	21 000	145	.8	16
	1900	1038	10 000	68.9	93.1	1.5
TAZ-8A						
As-extruded	1200	649	105 000	724	374.3	7
	1900	1038	[a]1 000	6.89	[b]>4.1	>600
	2000	1094	[a]2 020	13.9	[b]>.1	>285
Heat treatment Y	1900	1038	15 000	103	0.6	16
Heat treatment X	1200	649	105 000	724	974.6	2
	1800	982	21 000	145	1.8	7
Heat treatment Q	1900	1038	15 000	103	2.2	-----

[a]Tests conducted by independent laboratory.
[b]Specimen did not break.

are listed in tables IV and V. Generally, only single tests were run at a particular test condition due to the limited amount of extruded powder product available. Both conical head and threaded head type cylindrical specimens were used. All test specimens were machined with a gage section 0.250 inch (0.6 cm) in diameter. The conical head specimens had a gage section 1.25 inch (3.2 cm) long and the threaded head specimen had a gage section 1.00 inch (2.54 cm) long. Elongation was determined by dividing total specimen elongation by the gage length. All tests, except as noted in tables IV and V, were performed at Lewis Research Center. All tests were run in accordance with recommended ASTM practice except in those cases where excessive elongation allowed the specimens to stretch beyond the uniform temperature zone of the furnace.

Metallography

Metallographic studies were made for both alloys. Photomicrographs are presented for the as-cast condition, the as-received powder, the as-extruded powders, and various heat treatments. The as-extruded and heat-treated powder product specimens, as well as the as-cast samples of each alloy, were etched electrolytically with a solution of 20 parts water, 20 parts glycerine, 10 parts concentrated nitric acid, and 5 parts hydrofluoric acid. The as-received powders were swab etched with a solution of 92 parts hydrochloric acid, 3 parts nitric acid, and 5 parts sulphuric acid.

Workability Tests

A limited evaluation of the workability of the powder products was conducted by forging, hot rolling, and hot pressing. The tests and the results obtained are described in the RESULTS AND DISCUSSION section.

RESULTS AND DISCUSSION

Tensile Properties

Tensile properties of Alloy 713C and TAZ-8A are shown in figures 3 and 4, and a compilation of all tensile data is provided in table IV. The powder products for both alloys had higher tensile strengths than the as-cast materials up to at least 1400° F (760° C), but the powder products had lower strengths than the cast alloys in the 1800° to 2000° F (982° to 1094° C) temperature range. The extruded and subsequently heat-treated powder products for both alloys had tensile strengths generally intermediate between the as-extruded powder products and the as-cast material over the temperature range considered. The room-temperature tensile strength of the as-extruded powder product was much greater than that of the as-cast material for both alloys; 221 700 psi (1530 MN/m^2) against 123 000 psi (848 MN/m^2) for Alloy 713C and 228 000 psi (1570 MN/m^2) against 128 000 psi (882 MN/m^2) for TAZ-8A. This was true at 1200° F (649° C) as well, where the as-extruded powder product strength was 194 900 psi (1340 MN/m^2) for Alloy 713C and 218 200 psi (1510 MN/m^2) for TAZ-8A. These values compared to 126 000 psi (867 MN/m^2) and 128 000 psi (882 MN/m^2) for the as-cast versions of these alloys.

The ductility of the extruded powder product of both alloys increased sharply at the higher temperatures. At 2000° F (1094° C), Alloy 713C had a 45-percent elongation, and at 1800° F (982° C) TAZ-8A had more than a 450-percent elongation. Heat treat-

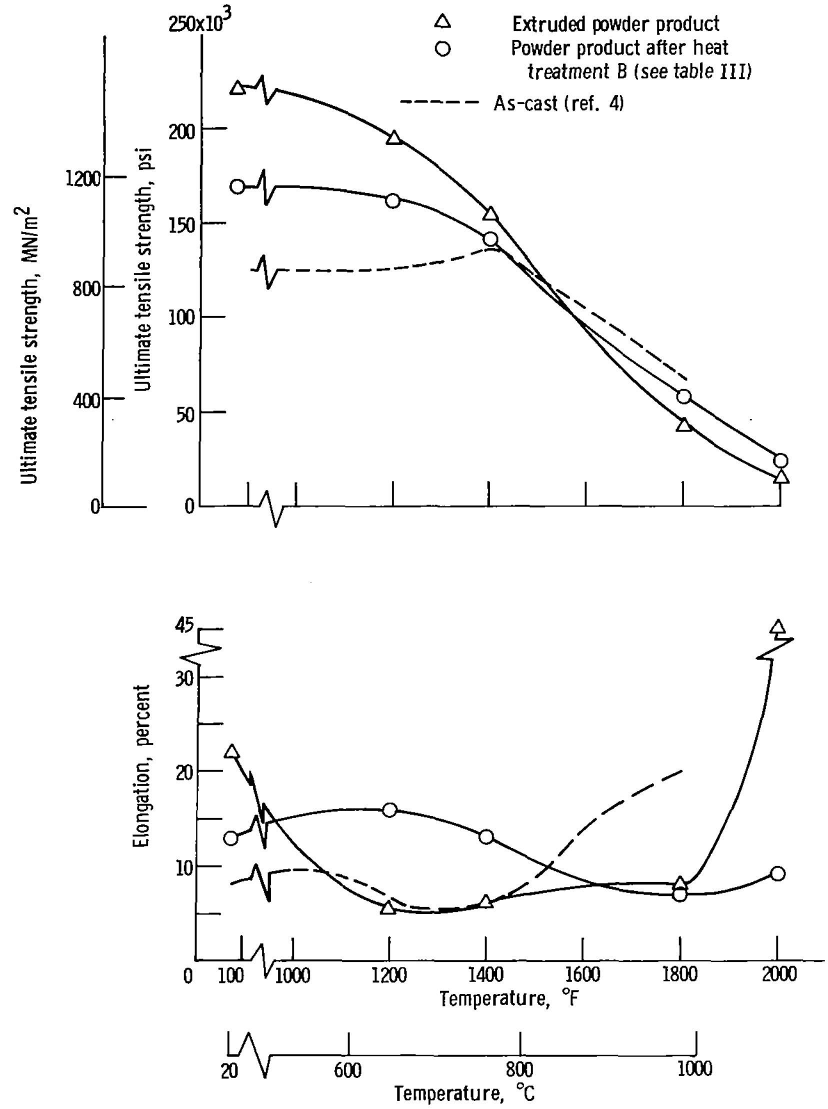

Figure 3. - Comparison of tensile properties of Alloy 713 C powder products and as-cast Alloy 713 C.

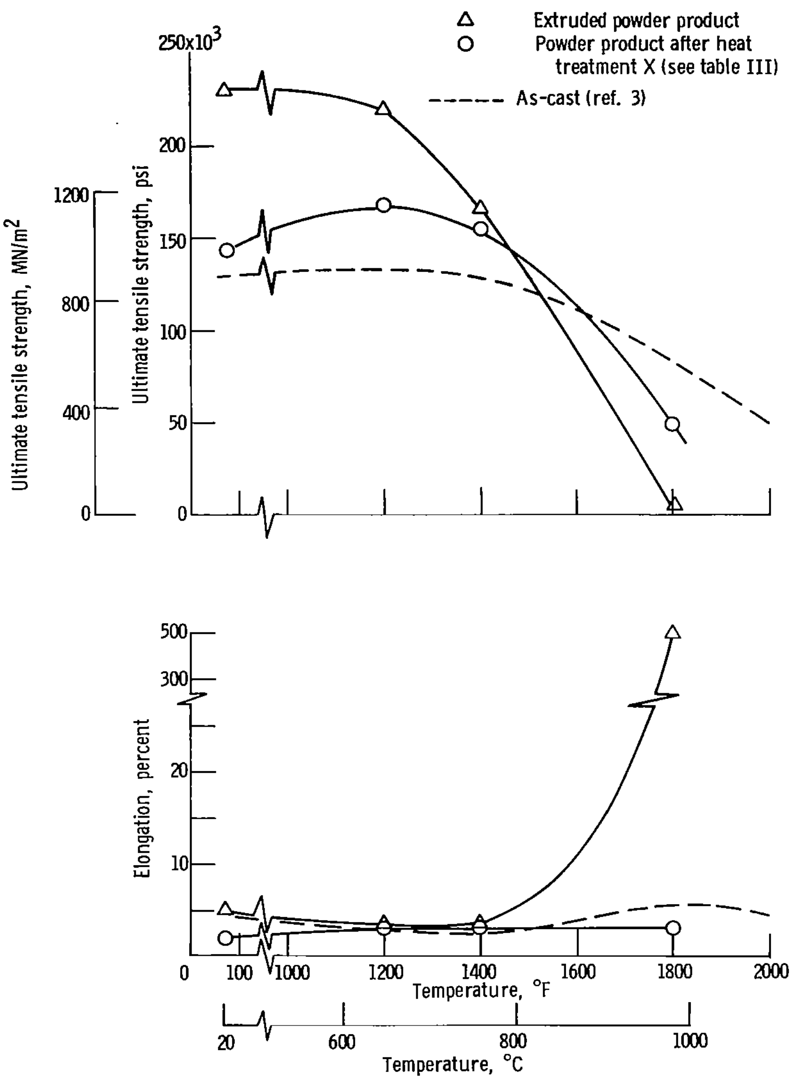

Figure 4. - Comparison of tensile properties of TAZ-8A powder products and as-cast TAZ-8A.

ments eliminated the tendency toward superplastic behavior (ref. 5) at high temperature on the part of the extruded powder products of both alloys. Also, the four-step heat treatment B (table III) given to Alloy 713C resulted in fairly high elongations ranging from 13 to 16 percent in the room to 1400° F (760° C) temperature range. Consideration of the elongation against temperature curves suggests that the as-extruded powders of both alloys may be amenable to working at temperatures of 1800° F (982° C) and above. In fact, earlier work (ref. 3) with conventionally cast TAZ-8A indicated that the alloy had a degree of workability potential in that cast slabs of the alloy could be rolled under closely controlled conditions. It should be noted, however, that the superplastic behavior in the powder product was observed at a relatively low stress. The high loads normally associated with large deformations in conventional working processes may therefore not be needed to utilize this superplastic behavior. This is, in fact, borne out by limited workability tests described in a subsequent section.

Stress Rupture Properties

The stress rupture data are summarized in table V. Figure 5 shows a comparison of the stress rupture life of the as-extruded and the extruded and heat-treated powder

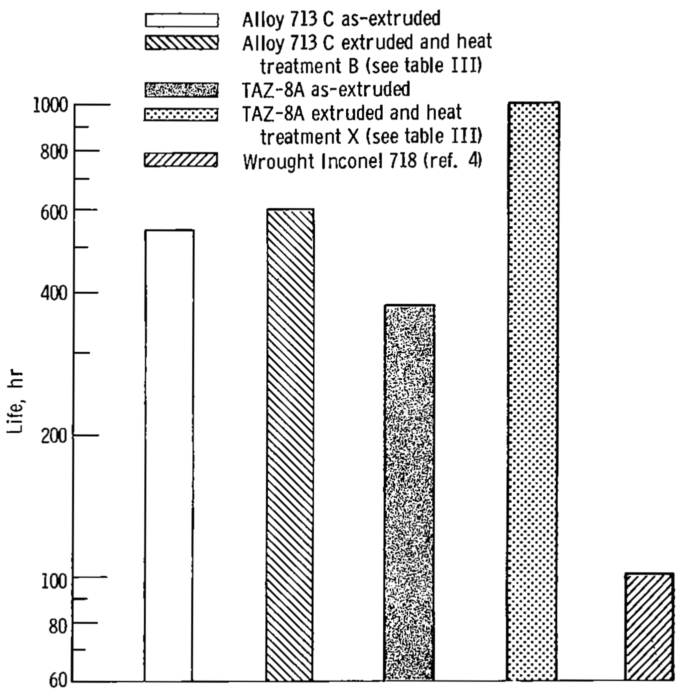

Figure 5. - Comparison of rupture lives of Alloy 713 C and TAZ-8A extruded and heat-treated powder products and conventionally wrought Inconel 718 at 1200° F (649° C) and 105 000 psi (725 MN/m²).

products of both alloys at 105 000 psi (724 MN/m^2) and 1200° F (649° C). Four-step heat treatments, which included aging at relatively low temperatures, were applied to both alloys. Rupture lives of the TAZ-8A powder products ranged from 374 hours for as-extruded material to 975 hours for extruded and heat-treated material. For Alloy 713C, these values were 538 and an average of 611 hours, respectively. For comparison, wrought Inconel 718, one of the stronger wrought nickel-base alloys for which data were available at 1200° F (649° C), has a life of 100 hours under these conditions. These favorable life properties suggest further that even at this early stage of development, the prealloyed powder product approach can give properties comparable to the more highly alloyed conventional wrought materials such as U-700. These comparisons are made to a wrought alloy to show the potential advantage of prealloyed powder products in applications such as the latter stages of turbine engine compressor disks where such wrought alloys are used. Of course, to utilize the prealloyed powder products in such applications, satisfactory forming techniques must be developed.

A comparison of stress rupture properties at 1900° F (1038° C) is made in figures 6 and 7. The as-extruded Alloy 713C powder product and the extruded material after be-

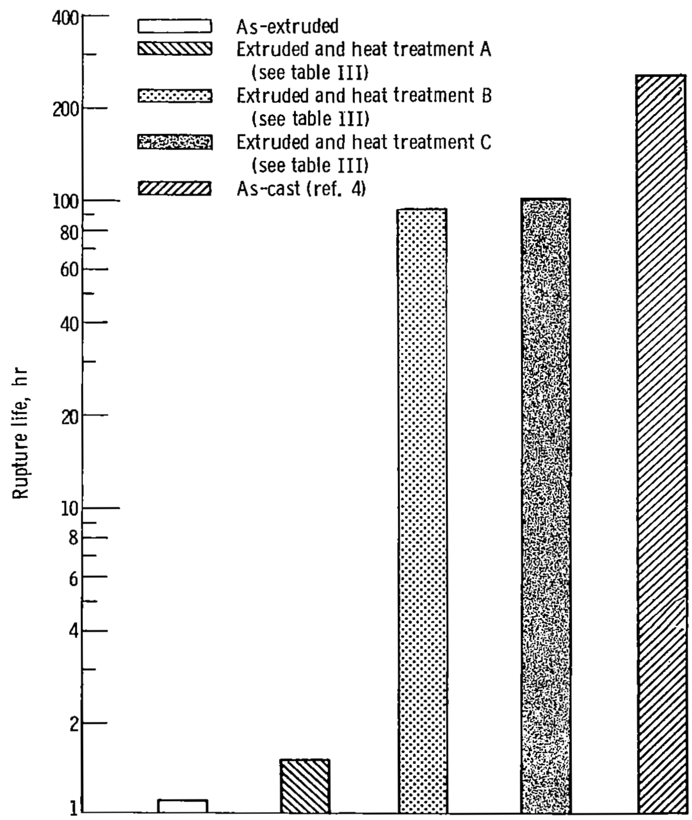

Figure 6. - Comparison of 1900° F (1038° C), 10 000 psi (68.9 MN/m^2) rupture life for extruded and heat-treated Alloy 713 C powder products and as-cast Alloy 713 C.

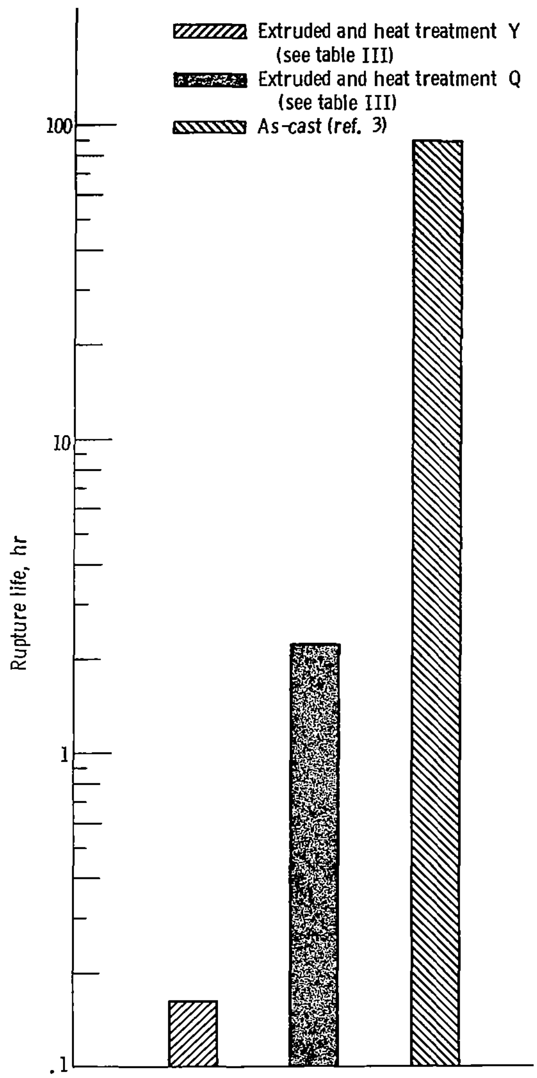

Figure 7. - Comparison of 1900° F (1038° C), 15 000 psi (103 MN/m^2) rupture lives for extruded and heat-treated TAZ-8A powder products and as-cast TAZ-8A.

ing subjected to various heat treatments are compared with as-cast Alloy 713C in figure 6. The longest life for the powder product, 98.1 hours, was obtained with a single high-temperature heat treatment (treatment C, table III). This life was still considerably less than the 250-hour, as-cast life. The as-extruded Alloy 713C had a life of only 1.2 hours and an elongation of 60 percent (table V).

It is interesting that similar improvements over the as-cast alloy in high-temperature stress rupture properties for Alloy 713C powder product to those reported in reference 1 were not obtained in the present investigation. Differences in test techniques and prealloyed powder manufacture and compaction could account for this lack of agreement. This suggests further that environmental control and quality control may be

As machined

After 4.1 hours at 1900° F (1038° C) and 1000 psi (6.89 MN/m^2); elongation, >600 percent

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Figure 8. - Stress rupture specimen of as-extruded TAZ-8A powder product before and after testing at 1900° F (1038° C).

extremely critical in the application of prealloyed powder techniques in order to achieve consistently reproducible results.

Figure 7 compares the rupture lives of the TAZ-8A heat-treated powder products with that of the as-cast alloy at 1900° F (1038° C) and 15 000 psi (103 MN/m^2). As was true for Alloy 713C, although the heat-treated TAZ-8A powder product had greater high-temperature rupture strength than the as-extruded material, its life was much lower (2.2 against 90 hr) than that of the as-cast alloy. High-temperature heat treatments (Y and Q, table III) intended to coarsen the microstructure and cause it to approach that of the as-cast material were applied to the TAZ-8A powder product. When it was attempted to test the as-extruded powder product at 1900° F (1038° C) and 15 000 psi (103 MN/m^2), the specimen stretched so fast that the creep machine could not accommodate the strain. It was therefore reloaded to a stress of 1000 psi (6.89 MN/m^2). This resulted in more than a 600-percent elongation after 4.1 hours (table V). Figure 8 shows this test specimen before and after test. Such superplastic behavior was eliminated by the heat treatments applied, and the high-temperature rupture strength was increased. Further heat-treating studies are needed to achieve a suitable microstructure for improved high-temperature properties. The effects of the various heat treatments on microstructure are discussed in the subsequent section on metallography.

Workability

Forging. - Attempts were made to hammer forge extruded bars of both alloys 6 inches (15.2 cm) long by 9/16 inch (1.4 cm) in diameter. The bars were first heated to 2200° F (1204° C) and then transferred as rapidly as possible to the forge. The Alloy 713C bar developed severe cracks after reduction to approximately a 0.45-inch (1.04-cm) thickness. The TAZ-8A bar shattered under the first blow. This work was done by the supplier of the powders.

Rolling. - The as-extruded TAZ-8A powder product was also evaluated for workability potential by rolling at the Lewis Research Center. Disks 1/4 inch (0.64 cm) in thickness were cut from as-extruded 9/16-inch- (1.4-cm-) diameter bars of TAZ-8A. Attempts were made to hot roll these disks at several temperatures from 1900° to 2100° F (1038° to 1149° C). A two-high rolling mill with 8-inch- (20.3-cm-) diameter rolls was used. Roll speed was approximately 80 feet per minute (41 cm/sec). Various reductions per pass were attempted. The smallest was 0.002 inch (0.005 cm). Despite the superplastic properties of the alloy at these temperatures, the disks cracked after only a few passes. The powder product was thus not amenable to forming at high strain rates.

Hot pressing. - A third approach taken to work the TAZ-8A powder product was by hot pressing. This also was done at the Lewis Research Center. A hydraulically operated press with a graphite susceptor-induction heating furnace was used. Specimens approximately 9/16 inch (1.4 cm) in diameter and 5/8 inch (1.6 cm) high were heated to 2000° F (1094° C) and pressed. Pressure was applied to the circular ends of the specimens through high-temperature alloy plates (TAZ-8B) which were heated to the same temperature as the specimen. An initial load of 155 pounds (690 N) was applied. Load was increased to maintain a relatively constant low strain rate of between 0.03 to 0.07 inch per inch per minute. This strain rate was set to approximate the average rate obtained when superplasticity was encountered with the alloy in stress rupture tests. The operation was interrupted to examine the specimen after the height of the specimen had been reduced by 50 percent. Since no cracks were evident, the specimen was returned to the press and a total reduction in height of 75 percent was obtained. As the specimen diameter increased, the limited capacity of the press prevented maintenance of the desired strain rate, and it dropped to less than 0.01 inch per inch per minute. This effectively limited the total reduction possible in this unit. Figure 9 shows the pressed

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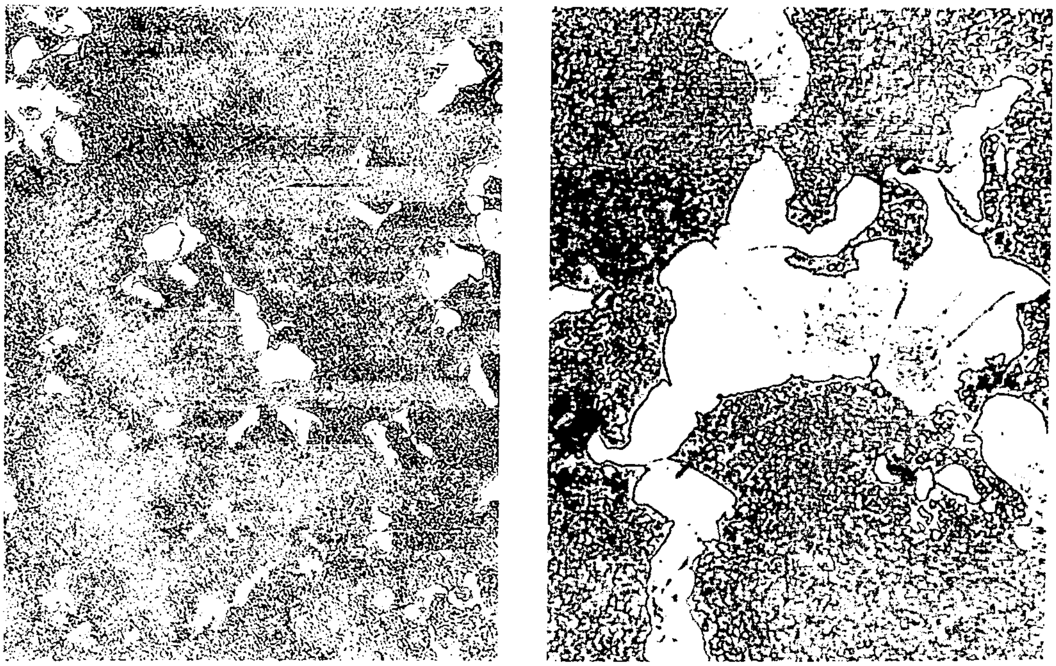
Figure 9. - Sample of TAZ-8A extruded powder product after upsetting at 2000° F (1094° C).

specimen and the starting blank. No cracks are evident around the circumference of the pressed piece. After pressing, the specimen had a diameter of 1.1 inch (2.8 cm) and its thickness was 0.175 inch (0.44 cm).

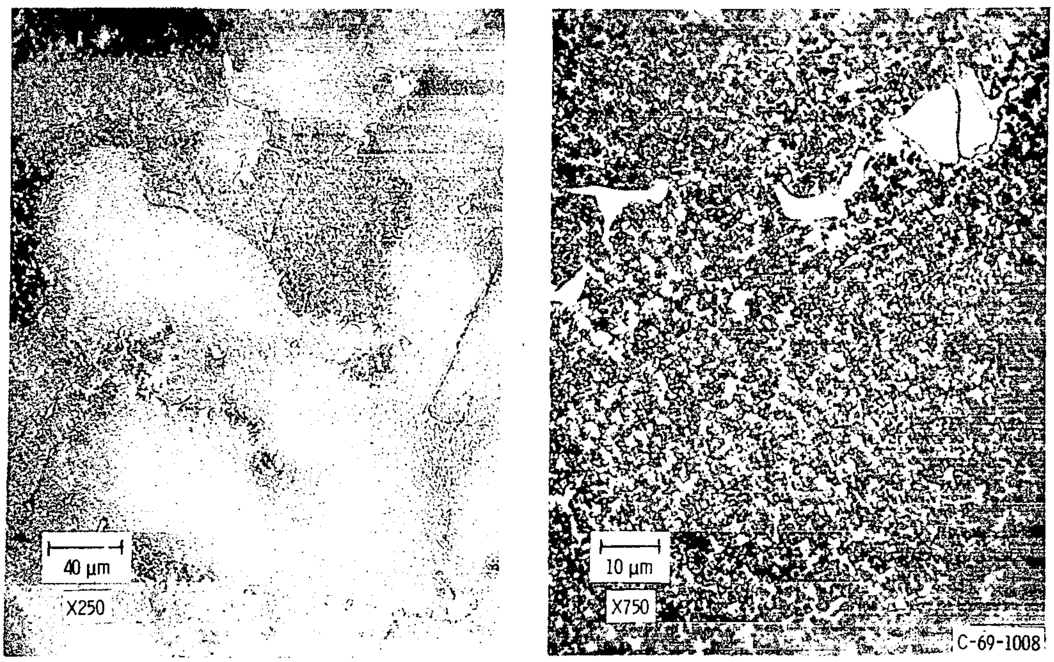
It is encouraging that the superplasticity observed in tensile tests in the as-extruded powder product can be used to form material in compression as well. The results of this small-scale evaluation suggest that larger disks could be hot pressed from prealloyed powders with relatively small presses and with the expenditure of relatively small amounts of power. Further work is, of course, needed to fully establish the effect of the various variables associated with forming superplastic powder products.

Metallography

As a frame of reference the microstructure of investment cast Alloy 713C and TAZ-8A are shown at 250x and 750x in figure 10. Both alloys have a matrix of nickel solid solution with a fine, barely resolved, precipitate of gamma-prime. Coring in the matrix is evident from the distribution of the gamma-prime precipitate. The chief dif-

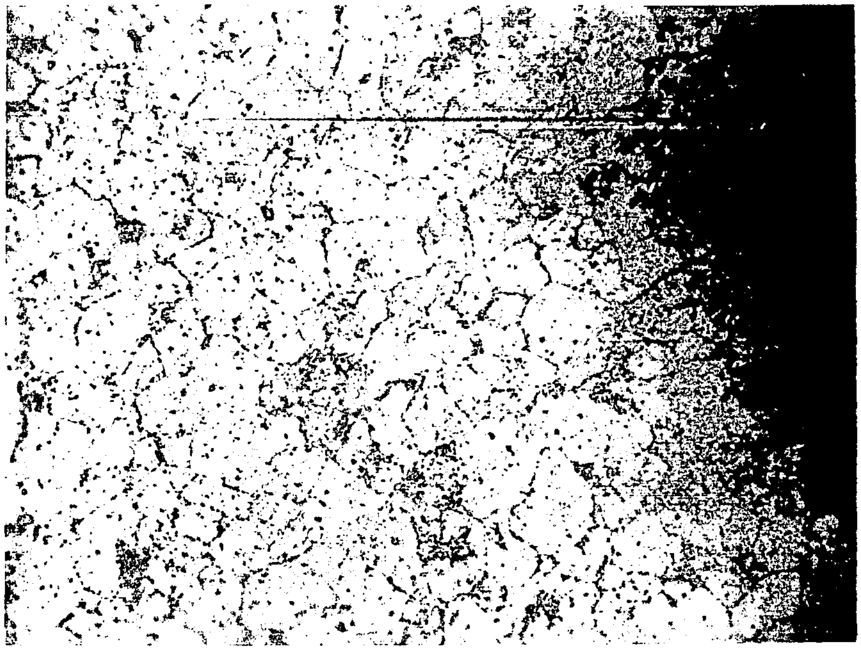


TAZ-8A

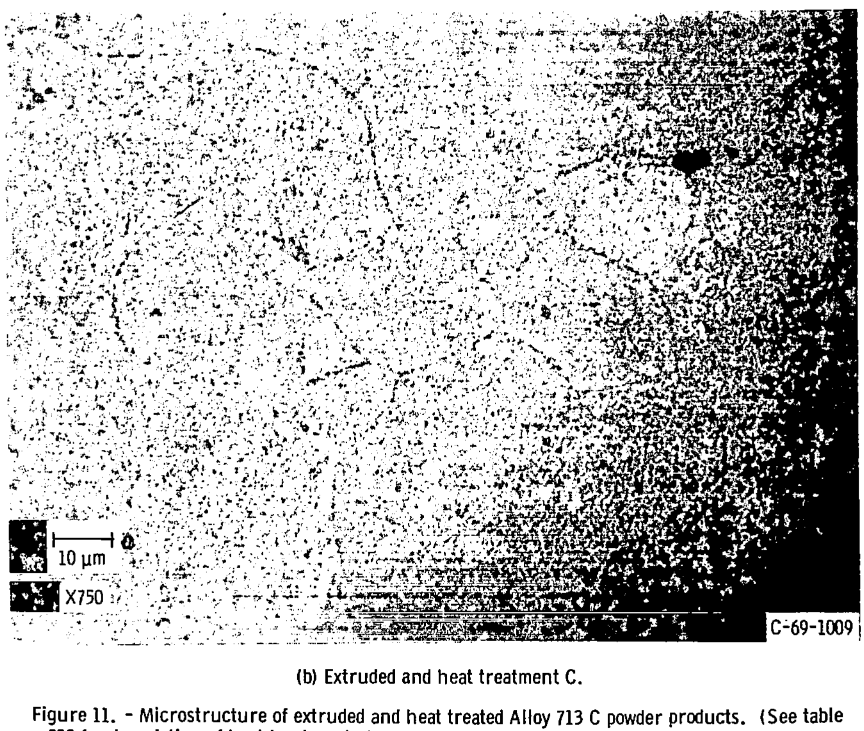


Alloy 713 C

Figure 10. - Microstructure of as-cast alloys.

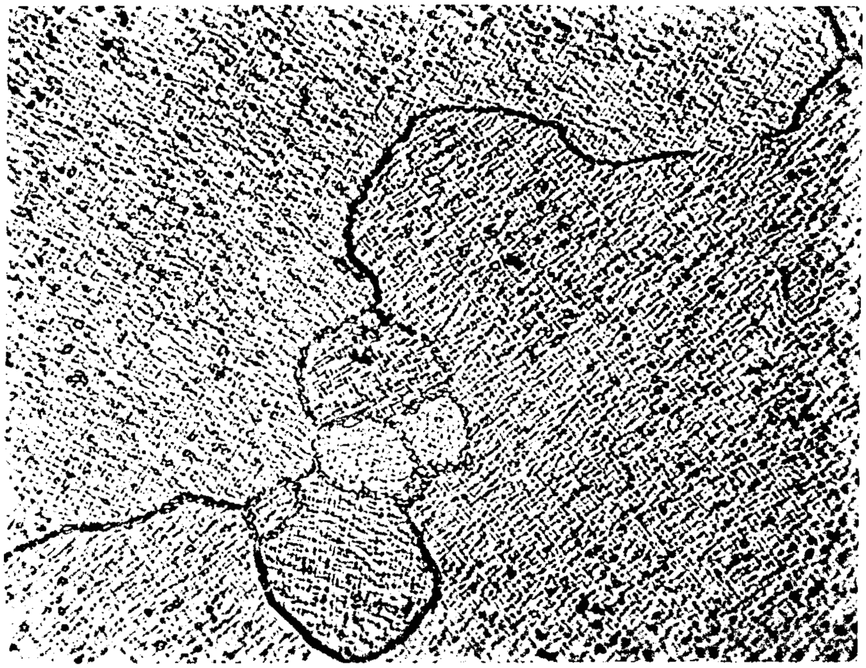


(a) As-extruded.

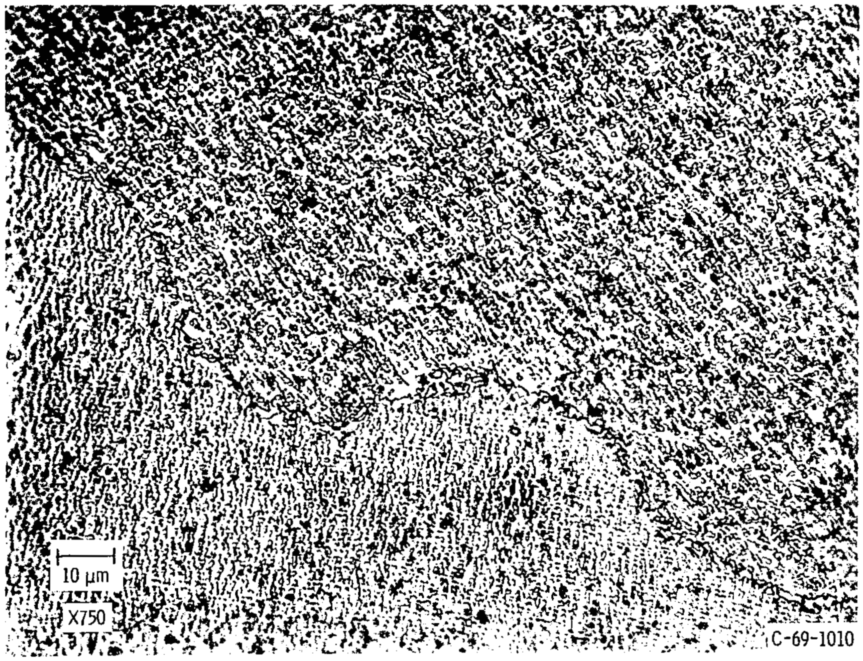


(b) Extruded and heat treatment C.

Figure 11. - Microstructure of extruded and heat treated Alloy 713 C powder products. (See table III for description of heat treatments.)



(c) Extruded and heat treatment B.



(d) Extruded and heat treatment A.

Figure 11. - Concluded.

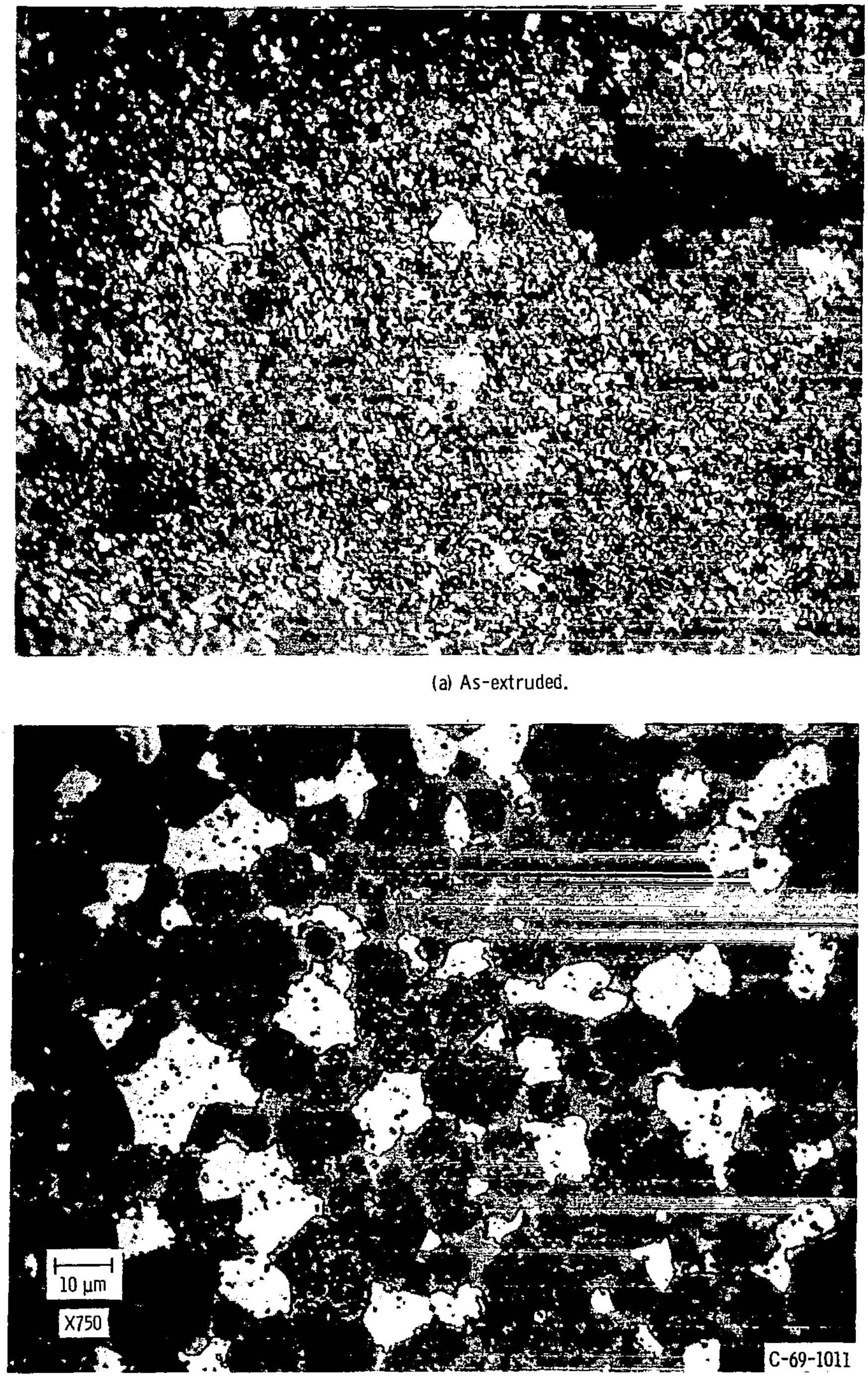
ference in the structures is the much larger amount of primary gamma-prime present in TAZ-8A. Interdendritic and grain boundary carbides are evident in Alloy 713C.

The powder particles of both alloys (fig. 1) exhibited a dendritic structure as described previously. Comparison with the conventionally cast structures in figure 10 makes it clear that the interdendritic spacing and hence the scale of microsegregation in the powder is at least an order of magnitude finer than in the conventional casting. This, of course, is one of the chief benefits of the atomized prealloyed powder approach. The largest single constituents present in the powders were a few idiomorphic MC carbides in the TAZ-8A powder.

Alloy 713C powder product. - Figure 11 shows the microstructure of Alloy 713C powder product in the as-extruded condition and after exposure to various heat treatments. The as-extruded Alloy 713C powder product (fig. 11(a)) had a fine-grained equiaxed structure. Although not completely resolved at this magnification, there was evidence of a dark etching precipitate at the grain boundaries and to a lesser extent within the grains. The gamma-prime precipitate in the grains is too fine to be resolved in this figure. In order to coarsen the structure for improved high-temperature properties, solution treatments at increasingly higher temperatures were attempted up to 2250° F (1232° C). Figure 11(b) shows the structure of the extruded Alloy 713C powder product after heat treatment C, 4 hours at 2250° F (1232° C) followed by a furnace cool. Even after exposure to this high temperature, the fine grains apparent in the as-extruded condition were not completely eliminated. A duplex grain size is clearly evident. There is also evidence that a minute amount of incipient melting occurred. The gamma-prime precipitate in the matrix was slightly coarser than in the as-extruded condition, and the grain boundary precipitate was more clearly defined.

The use of the four-step heat treatment B (table III) resulted in the structure shown in figure 11(c). This is similar to the multiple-step heat treatments applied to various nickel-base alloys (ref. 4). It is believed that the 2000° F (1094° C) exposure was responsible for coarsening the gamma-prime precipitate as well as for the heavier precipitate observed at the grain boundaries. Again a duplex grain size is clearly evident. A similar heat treatment (heat treatment A) involving the same four steps produced the structure shown in figure 11(d). Heat treatment A differed from B only in that the specimens were furnace cooled instead of air cooled from 2250° F (1232° C). The structures are very similar except that the furnace-cooled structure is slightly coarser. Although it is not apparent from this figure, a duplex grain size occurred.

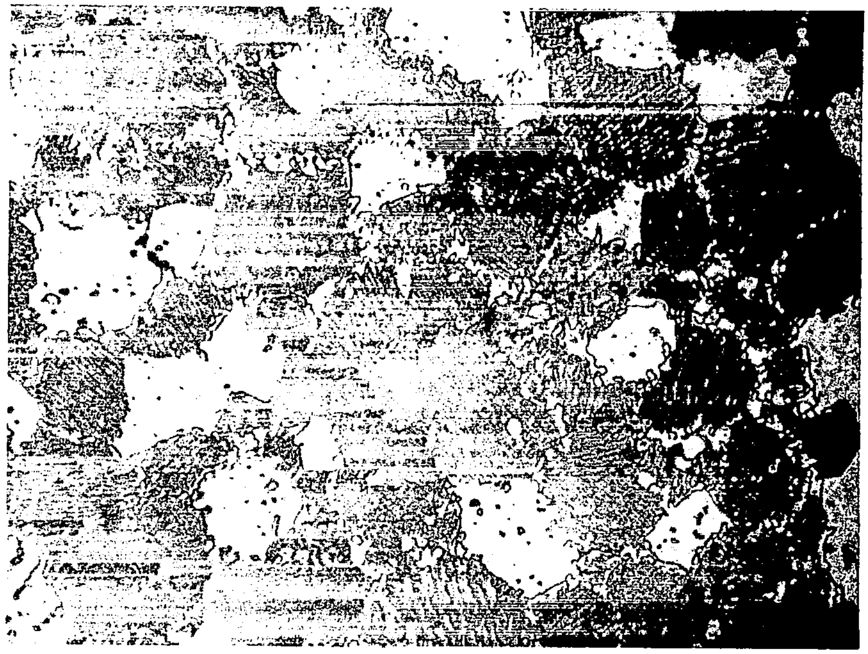
TAZ-8A powder product. - The microstructures of TAZ-8A powder product in the as-extruded condition and after various heat treatments are shown in figure 12. Figure 12(a) shows as-extruded material. Minute grains of gamma and gamma-prime are evident. This section was taken near the center of a portion of a bar showing radiographic evidence of internal voids. The voids appear to have been the result of tearing rather than local melting due to overheating during extrusion and may be due to center burst



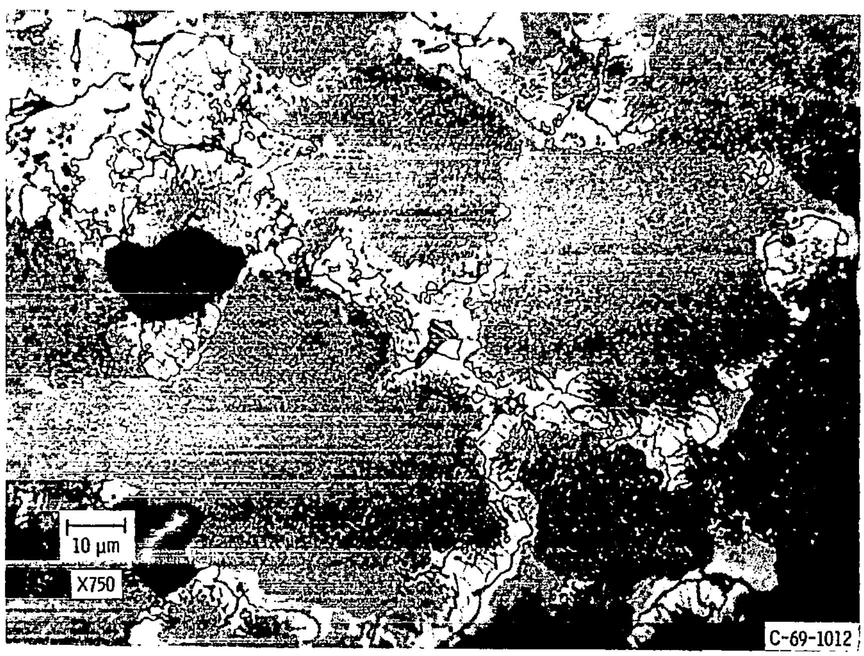
(a) As-extruded.

(b) Extruded and heat treatment Y.

Figure 12. - Microstructure of extruded and heat treated TAZ-8A powder products. (See table III for discription of heat treatments.)

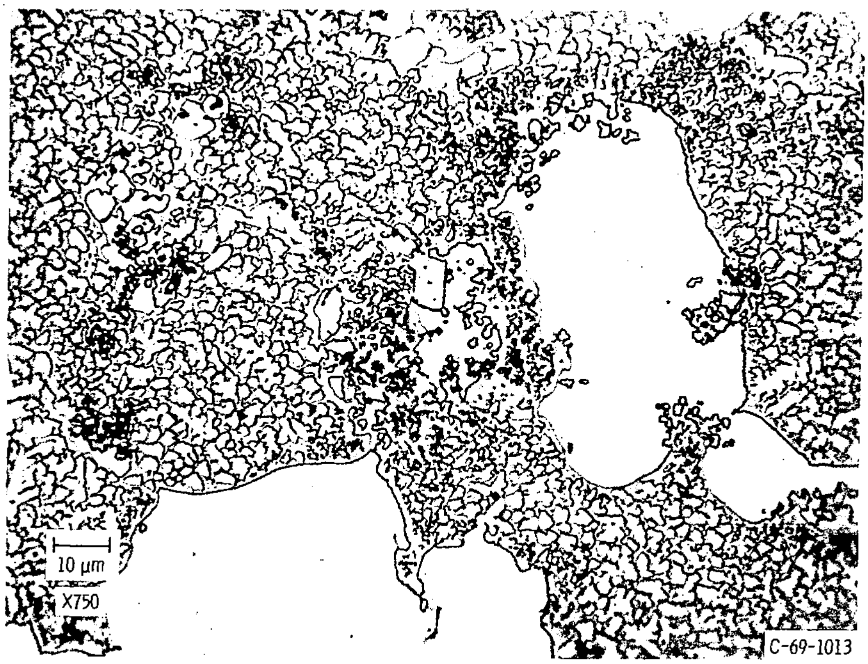


(c) Extruded and heat treatment X.



(d) Extruded and heat treatment Z.

Figure 12. - Continued.



(e) Extruded and heat treatment Q.

Figure 12. - Concluded.

during extrusion. In order to coarsen the structure, increasingly higher solution treating temperatures were attempted. It was possible to apply a 2350° F (1288° C) temperature without encountering significant minor phase melting. The microstructure of figure 12(b) resulted from exposing the extruded powder product for 1/2 hour at 2350° F (1288° C) and air cooling (heat treatment Y). Despite the high-temperature exposure, there was no substantial solutioning of the massive gamma-prime particles. These appear somewhat like the primary gamma-prime in the cast alloy but are more equiaxed. They are more polygonal, are less associated with grain boundaries, and contain isolated precipitate particles rather than a precipitate network. The matrix (or perhaps more correctly, the other constituent in this duplex structure) was gamma and it contained a fine precipitate of gamma-prime.

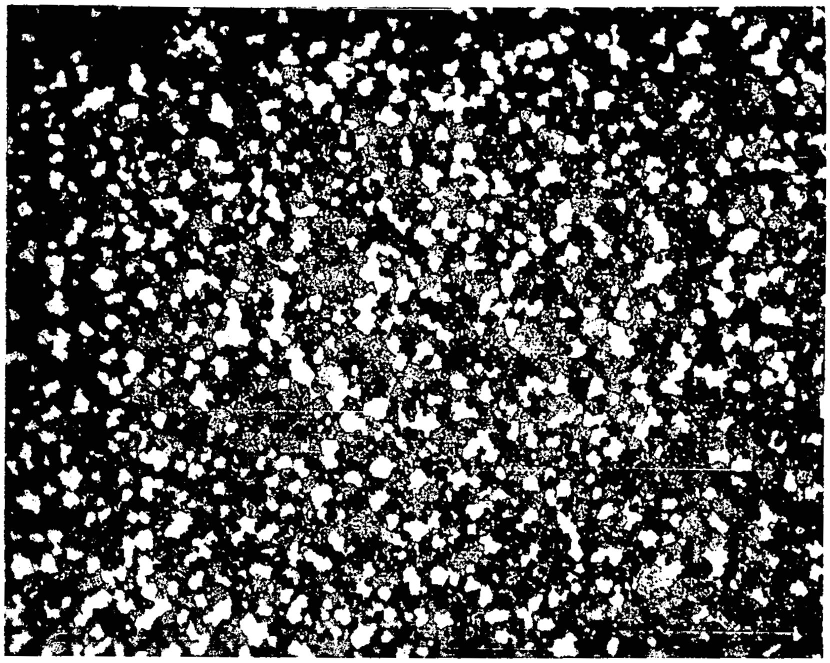
The same three-step aging treatment used for Alloy 713C was applied to TAZ-8A "solution" treated from 2350° F (1288° C). The microstructure resulting from this heat treatment X is shown in figure 12(c). The heat treatment coarsened the precipitate in the gamma and also produced a grain boundary precipitate. The apparent increase in grain size shown in figure 12(c) as compared to that in figure 12(b) may be due at least in part to the 2000° F (1094° C) first step age.

In order to coarsen the structure of TAZ-8A powder product further, some heat treatments were attempted substantially above the incipient melting temperature. An example of the structure resulting from such a heat treatment (heat treatment Z), is shown in figure 12(d). It is apparent that exposure for 1/2 hour at 2400° F (1316° C) caused severe incipient melting. The gamma-prime phase also appeared to have melted and taken on an internal structure typical of so-called eutectic gamma-prime. Instead of being present as the distinct grains which formed at 2350° F (1288° C), gamma-prime now appeared as a grain boundary constituent. The matrix was gamma with a fine precipitate of gamma-prime. Considerable grain coarsening was observed; however, a substantial number of voids formed due to incipient melting.

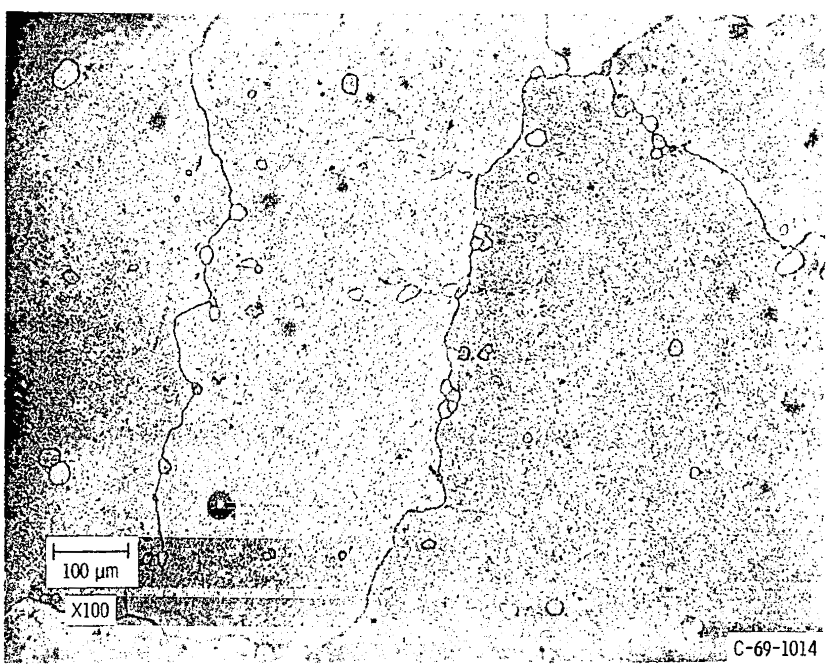
To achieve coarsening without void formation, specimens were subjected to pressure during exposure to temperatures above the incipient melting point. They were heat treated in an autoclave under helium pressure. The microstructure of a specimen exposed at 2400° F (1316° C) and 10 000 psi (68.9 MN/m^2) for 2 hours, heat treatment Q, is shown in figure 12(e). No voids were observed. The specimens heat treated in the autoclave were furnace cooled. This contributed to the much coarser precipitate of gamma-prime in the gamma matrix as compared to figure 12(d). Most important, the application of a relatively low pressure (10 000 psi (68.9 MN/m^2)) prevented the formation of the voids so evident in specimens heated under atmospheric pressure. Although the microstructure showed no obvious evidence of melting, the exterior of the autoclave specimen showed the same characteristic-dished ends and prune-skin surface found on specimens heat treated under atmospheric pressure. This indicates some incipient melting had indeed occurred. This surface layer can be removed, however, leaving an internally sound specimen.

The autoclave heat treating results are encouraging even though only very limited data were obtained. Somewhat better stress rupture properties were obtained than from standard heat treatments. They suggest further that by a combination of autoclave and subsequent atmospheric pressure heat treatments it may be possible to achieve structures which will have elevated temperature (1800° F, 982° C and above) properties as good or better than cast material. It may be that the autoclave could also be used for simultaneously consolidating prealloyed powders into turbine blade shapes and imparting structures suitable for high-temperature service.

An important aspect of the metallurgy of TAZ-8A insofar as making a satisfactory high-strength powder product has to do with coarsening its grain structure. Even though the size of the gamma and gamma-prime constituents of the powder product (fig. 12(e)) were coarsened to a size similar to that observed in the as-cast structure (fig. 10), the grain size was not increased correspondingly. For example, a cast tensile specimen of TAZ-8A had about 10 grains across the cross-sectional area of the 1/4-inch test section. The scale of the grains in the heat-treated powder product, on the other hand, is on the same order as the size of the gamma-prime constituents (fig. 12(c)) which are roughly 0.001 inch (0.0025 cm) in diameter. The comparative grain size of the TAZ-8A and Alloy 713C powder products after heat treatment, is shown in figure 13 at 100 X.



(a) TAZ-8A extruded plus heat treatment X.



(b) Alloy 713 C extruded plus heat treatment C.

Figure 13. - Grain size and microstructure of heat treated Alloy 713 C and TAZ-8A extruded powder products. (See table III for description of heat treatments.)

The grain structure of Alloy 713C was more easily coarsened by heat treatment because all of the gamma-prime phase could be taken into solution. But, as can be seen from figure 13(b), even for Alloy 713C, a number of small grains persisted after heat treating at 2250° F (1232° C). It is believed that the application of pressure at high temperature for longer time periods than have been attempted thus far, will enhance the possibility of achieving the desired larger grain structure in TAZ-8A. This problem of achieving a sufficiently coarse grain structure may be expected to prove equally difficult with other highly alloyed nickel-base systems.

CONCLUDING REMARKS

Although only limited data have been obtained thus far, the present investigation indicates that the use of prealloyed powders as a means of producing high-strength, nickel-base alloys has considerable potential. Significant increases were observed in intermediate temperature strength with the prealloyed powder product as compared to as-cast properties for both alloys investigated. The demonstrated high ductility of the as-extruded powders in the 1800° F (982° C) to 2000° F (1094° C) range suggests that these materials may be amenable to hot-forming operations. The superplastic behavior observed in tensile and stress rupture tests can be utilized by hot pressing at low loads and low strain rates to readily form these materials. The excellent 1200° F (649° C) properties of the powder products indicates a great potential for prealloyed superalloy powder products in such applications as the compressor disks of the high-temperature stages of advanced engines. Further investigation is required to achieve high strength at more elevated temperatures.

The metallographic results of this investigation together with those of earlier investigators indicate that macro- and micro-segregation which can be encountered in conventional castings can be eliminated by the use of prealloyed powders. This suggests that alloys can now be designed to contain higher quantities of strengthening alloying constituents than can be accommodated by conventional casting. This, in turn, could lead to a higher level of strength capability for superalloys than is currently envisioned for this class of material. Of course, it must be realized that this approach is not without its problems. In particular, methods of achieving satisfactory microstructures for high temperature application must be developed.

SUMMARY OF RESULTS

Evaluation of prealloyed powders of two nickel-base superalloys, Alloy 713C and

TAZ-8A, in the as-extruded and extruded-and-heat-treated condition provided the following major results:

1. In the as-extruded condition, the powder products of both alloys had very high elongations in elevated temperature tensile and stress rupture tests, suggesting superplastic behavior. For example, Alloy 713C had a rupture elongation of 230 percent after testing at 2000° F (1094° C) and 2000 psi (13.8 MN/m^2) for 7.6 hours and TAZ-8A had an elongation of more than 600 percent after testing at 1900° F (1038° C) and 1000 psi (6.89 MN/m^2) for 4.1 hours.

2. It was shown that it is possible to take advantage of the superplastic behavior of as-extruded prealloyed powder products by hot pressing. Samples of TAZ-8A about 5/8 inch (1.6 cm) high by 9/16 inch (1.4 cm) in diameter were upset without cracks at 2000° F (1094° C) at low strain rates of 0.03 to 0.07 inch per inch per minute and under relatively low loads (initial load applied was 155 lb, 690 MN). Height reductions of 75 percent were achieved.

3. The room-temperature tensile strength of the as-extruded powder products for both alloys was substantially greater than that of conventionally cast or wrought nickel-base superalloys. Ultimate tensile strengths were 221 700 psi (1530 MN/m^2) and 228 000 psi (1570 MN/m^2) for Alloy 713C and TAZ-8A, respectively. This compares with 123 000 psi (846 MN/m^2) and 128 000 psi (882 MN/m^2), respectively, for these alloys in the as-cast condition.

4. The superiority in tensile strength of the as-extruded powder products over the as-cast material was maintained for both alloys up to 1400° F (760° C), but the powder products had lower strengths at temperatures of 1800° to 2000° F (982° to 1094° C), respectively. Extruded and subsequently heat-treated powder products of both alloys had tensile strengths intermediate between those of the as-extruded powder product and the as-cast material over the entire temperature range.

5. Heat treatments, which coarsened the microstructure of the extruded powder products, substantially improved stress rupture life for both alloys compared to the life of the as-extruded powder products at all temperatures investigated. At 1200° F (649° C) and 105 000 psi (724 MN/m^2), the extruded and heat-treated powder products of Alloy 713C and TAZ-8A had rupture lives of 611 and 975 hours. This compares to 100 hours for Inconel 718, one of the stronger conventionally wrought nickel-base alloys available.

6. Heat-treated extruded powder products of both alloys had substantially lower rupture lives at high temperatures (1800° to 2000° F, 982° to 1094° C) than in the as-cast condition. For Alloy 713C, the life comparison was 90 against 250 hours at 1900° F (1038° C) and 10 000 psi (68.9 MN/m^2). For TAZ-8A, it was 2.2 against 90 hours at 1900° F (1038° C) and 15 000 psi (103.2 MN/m^2).

7. By simultaneously applying pressure and temperature, it was possible to exceed the incipient melting point (at 1 atm) of as-extruded TAZ-8A alloy powder product without void formation. As-extruded TAZ-8A powder was successfully heated to 2400° F

(1316° C), about 50° above the incipient melting point, under a pressure of 10 000 psi (68.9 MN/m^2). By this technique, it is possible to coarsen the microstructure to a greater degree than by conventional heat treatments.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 4, 1969,
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